SPACE SCIENCES LABORATORY

FINAL REPORT

NASA Grant NGR 05-003-512

The Study of Electrical Conduction Mechanisms

Principal Investigator
Professor H. F. Morrison

Scientific Collaborator
Dr. R. Alvarez

Period of Performance
1 February 1973 - 31 January 1974

April 18, 1974

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UNIVERSITY OF CALIFORNIA, BERKELEY

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BIBLIOGRAPHY

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This report is divided into two parts. The first describes the analysis of the dielectric variations of lunar fines sample 74241,2. The second gives a brief description of the electrical properties of solid sample 70215,14.

PART I

Dielectric Comparison of Lunar and Terrestrial Fines at Lunar Conditions

ABSTRACT

The dielectric response of lunar fines 74241,2 is presented in the audio-frequency range and under lunarlike conditions. Results suggest that volatiles are released during storage and transport of the lunar sample. Apparently, subsequent absorption of volatiles on the sample surface alter its dielectric response. The assumed volatile influence disappear after evacuation. A comparison of the dielectric properties of lunar and terrestrial materials as a function of density, temperature, and frequency indicates that if the lunar simulator analyzed were completely devoid of atmospheric moisture it would present dielectric losses smaller than those of the lunar sample. It is concluded that density prevails over temperature as the controlling factor of dielectric permittivity in the lunar regolith and that dielectric losses vary slowly with depth.

INTRODUCTION

The process of data acquisition on the electrical properties of returned lunar materials encompasses a period of approximately four years. The initial measurements were performed at high and audio-frequencies (Gold et al., 1970; Chung et al., 1970); little attention was paid at this stage to the effect of atmospheric water contamination on the samples' electric and dielectric response. Studies on terrestrial materials suggested that minute amounts of atmospheric moisture should have large effects on the electrical properties of lunar samples (Alvarez, 1971); subsequent experiments proved that such an effect was indeed observed in lunar materials (Strangway et al., 1972). As a result of these experiments it is now recognized that electric and dielectric measurements have to be carried out under controlled atmospheres (e.g., high vacuum) in order to obtain values representative of the materials in situ. On the basis of results presented herein it is suggested that volatiles other than H_2O may also affect the dielectric properties of lunar samples.

The bulk of the dielectric data available to date consists of measurements at frequencies below 10 MHz; in such a frequency range, experimental conditions have varied from nitrogen atmospheres at room temperature (Katsube and Collett, 1971 and 1973) to nitrogen atmospheres at various temperatures (Chung et al., 1970 and 1971) and to high vacuum and temperatures ranging from 77°K to 773°K (Chung et al., 1972 and 1973; Strangway et al., 1972; Olheft et al., 1972, 1973a and 1973b). Dielectric data is also available at substantially higher frequencies (Gold et al., 1970, 1971, 1972, and 1973; Bassett and Shackelford, 1972). Some studies have focused on electrical conductivity at various temperatures and atmospheres (Schwerer et al., 1971, 1972, and 1973).

Reproducing lunar surface conditions involves subjecting a sample to high vacuums and temperatures varying from 100° to 400°K approximately. The present study analyzes the dielectric variations of lunar sample 74241,2 under such conditons. The experimental technique and apparatuses used in these measurements are essentially those described by Alvarez (1973a), with the additional precaution of never exposing the lunar samples to terrestrial atmosphere. Handling of the sample (i.e., weighing, transferring to sample holder, etc.) was made in N_2 atmospheres of zero percent relative humidity, determined by a relative humidity meter.

Sample 74241,2 was collected in the South rim of Shorty Crater, at Station 4. The sample fraction analyzed is composed of grants of less than one millimeter.

Its composition is 60 percent basalt (no vugs), 20 percent basalt (vugs), 10 percent white dusty crystalline fragments, and 10 percent black glass fragments. Further description of the sample appears in Lunar Sample Info. Cat. (1973).

Samples of two packing densities were analyzed: $\rho = 1.38 \pm .05$ and $\rho = 1.61 \pm .05$ g/cm^3. The denser sample was obtained by placing the same amount of fines available (i.e., approximately 5 g) in a sample holder of smaller volume. Sample thicknesses were 1.4 and 1.1 mm respectively. Each sample was measured in the 30 Hz to 100 KHz frequency range at temperatures of 100°; 298°, and 373°K. The experimental error at all frequencies and temperatures for κ' is $\pm$ .01, and for tan δ it is $\pm$ .001 (at 30 Hz) or better, except for the 100 Hz value at room temperature and 2.0 torr pressure (Figure 1b) which has error limits of $\pm$ .005.

Dielectric Response Of Sample 74241,2

The dielectric permittivity (κ') and loss tangent (tanδ) data for the sample of density 1.38 g/cm^3 appear in Figures 1a and 1b respectively. The temperature sequence was as indicated in the figures. The first set of measurements, at room temperature and a pressure of 2 torr of N_2, can be regarded as representative of the sample properties "as received" (i.e., the sample had not previously been subjected to evacuation or temperature variations). According to the Lunar Receiving Laboratory, the sample was never exposed to the terrestrial atmosphere. For transportation the sample was packed in a stainless steel vial and three plastic bags filled with nitrogen; in-transit contamination between laboratories is thus regarded as improbable. As pointed out previously, the sample was handled in a zero percent relative humidity atmosphere of N_2 in our laboratory.

In spite of the precautions taken to avoid contamination, the dielectric response of the sample as received indicates that some sort of alteration occurred while in storage, transit, or handling. The κ' values at room temperature and 2 torr of N_2 (Figure 1a) show a marked tendency to increase towards the lower frequencies, while at frequencies above 1 KHz the response is practically the same as those at room temperature and high vacuum. The corresponding tan δ values (Figure 1b) also show large increments with respect to the high vacuum values for frequencies below 10 KHz. Alteration of the samples' dielectric properties is thus manifested at the low-frequency end of the frequency range analyzed.

After measuring at room temperature and 2.0×10^{-9} torr, the sample was cooled at 100°K at 2.5×10^{-9} torr. Lowering the temperature resulted in a slight decrease of the κ' values while the tan δ values experienced minor variations. Next, measurements at room temperature and 1.6×10^{-9} torr were repeated, closely reproducing the κ' and tan δ values previously obtained at such conditions. This sequence of results shows that: (1) evacuation of the sample changed its original (i.e., as received) dielectric response and, (2) the response at room temperature and high vacuum is reproducible. Further on, it will be shown that the as-received response is not recoverable after evacuation.

Evacuation could have only removed volatiles from the sample, consequently the dielectric response of the sample as received must have been influenced by such volatiles. The exact mechanism by which alteration of the electrical properties of the sample took place cannot be determined with the present information; however, two possibilities are offered: (1) solar wind trapped gases being released (e.g., while in transport) from original formation sites, with subsequent physisorption to grain surfaces or, (2) water vapor formation in the submonolayer region by a process similar to that suggested by Cadenhead et al., (1973).

In both cases active gases (e.g., oxigen, water vapor) would alter the surface conductivity of sample grains (Alvarez, 1973b), just as they do in the case of semiconductors and insulators (e.g., Buck et al., 1965) increasing the values of κ' and tan δ toward the low-frequency end. The results of Strangway et al., (1972) confirm that water contamination results in such increases in lunar fines. Further discussion on conductivity effects in dielectric measurements appears in Alvarez (1973c). In lunar samples there is no direct evidence of electrical alterations by active gases other than H_2O.

Figures 2a and 2b show κ' and tan δ responses for the 1.61 g/cm^3 density value. In order to obtain this new density the sample had to be re-packed in a thinner sample holder, as previously indicated. Such an operation involved transferring the sample from the high vacuum chamber to a nitrogen tent and back to the vacuum chamber. The steps followed were the same as when preparing the sample of density 1.38 g/cm^3, so that if contamination had occurred during the first manipulation it would also be expected to occur in the latter. This is pointed out to stress the fact that in Figures 2a and 2b, no low-frequency increases at room temperature and 1 torr of N_2 are observed and consequently no contamination resulted from such manipulations. In the present case there is excellent agreement between responses at room temperature and pressures of 1 torr and 2.0 x 10^{-9} torr. Lowering the temperature to 100°K and increasing it to 373°K resulted in variations of κ' and tan δ similar to those obtained in the previous case. Minor variations of tan δ values with temperature were obtained in each instance.

Lunar And Terrestrial Materials

The following is a comparison between the dielectric responses of a lunar and a terrestrial material as a function of density and with frequency and temperature as parameters. Results for the lunar material correspond to sample 74241,2 described in the present work. The terrestrial material was prepared to simulate lunar fines of Apollo 12; the corresponding results were previously reported (Alvarez, 1973a). Both sets of results were obtained at essentially the same vacuum and temperature conditions using the same experimental equipment and techniques. Thus, the comparison is considered to truly reflect common trends and differences between the dielectric behaviour of the two materials. Criteria for inferring lunar material responses from studies on terrestrial analogues can be established with such type of comparisons.

Figures 3a, 3b, and 3c show dielectric permittivity versus density curves for five density values and three temperatures. In each figure data are plotted for one of three frequencies: 1, 10, or 100 KHz. The three highest density values correspond to the terrestrial material and the two lowest ones to the lunar sample. κ' values for the lunar material show small variations with temperature at any frequency, whereas the κ' values of the terrestrial material experience large excursions as a function of temperature. It is readily observed that as the frequency increases the behaviour of the terrestrial material approaches that of the lunar sample, showing decreasing spread in the κ' values for a given density. In general, it can be seen that higher κ' values correspond to higher density values for a given frequency and a given temperature; this relation is better appreciated at the higher frequencies.

Figure 4 shows the tan δ curves corresponding to the values shown in Figures a, b, and c. The transition from lunar to terrestrial material is neatly shown. The same compactness observed for the κ' data of the lunar sample is again manifested in the tan δ data. The response of the terrestrial material spreads in a highly symmetrical fashion with respect to the lunar sample data. Tan δ values for the terrestrial sample show no clear tendency to approach lunar material behaviour with increasing frequency, contrasting with the observations made for κ' (Figure 3).

Of particular interest is to observe the smaller losses of the terrestrial material for temperatures of 100° and 298°K (except for the 1 KHz and 298°K curve). The observation appears to be in direct contradiction with logical expectations since, (1) both sample 74241,2 and the terrestrial material are essentially basalts, (2) densities for the latter are higher than those for the lunar sample and (3) the tan δ response of the terrestrial sample was affected by some atmospheric moisture, notwithstanding evacuation and heating (Alvarez, 1973a), which tends to increase the losses at room temperature.

The necessary conclusion is that the terrestrial material has smaller intrinsic losses (i.e., the losses of the water uncontaminated material) than the lunar sample. To substantiate this assertion, assume that the terrestrial material completely devoid of moisture would behave as the lunar sample (i.e., with small spreading of tan δ values as a function of temperature). The dielectric effects of small amounts of moisture on the sample's response are known to be minimal at temperatures of around 100°K (Alvarez, 1973d and 1973e). Thus, the tan δ values at 100°K and ultra-dried conditions would not be expected to depart much from the values presented in Figure 4 for such a temperature and 100 KHz. This curve could tentatively be considered as a base line above which tan δ increments would take place with increasing temperatures. If the assumption is correct, the tan δ response of the terrestrial material for the temperature range analyzed would lay below the values obtained for the lunar sample. There is no sufficient information to attempt an explanation of the basic mechanism responsible for the higher losses in the lunar material. Mineralogical composition certainly plays an important role in determining κ' and tan δ values; however, there is little information available on the role of specific minerals and the way they affect such values in lunar fines or powdered terrestrial materials.

DISCUSSION

The results presented on sample 74241,2 simulating lunar surface conditions show that the overall κ' variation from lunar night to lunar day is less than that produced by an increment in density of approximately 0.2 g/cm^3 (i.e., from 1.40 to 1.60 g/cm^3). According to Robie and Hemingway (1971) the synodic temperature variation is reduced to about 6°K at lunar surface depths of 20 to 30 cm. In addition, the results of Carrier et al. (1973) indicate that increments of around 0.3 g/cm^3 occur in the first 10 cm of the regolith (i.e., approximately from 1.40 to 1.70 g/cm^3). Thus, it is clear that in the regolith, density prevails over temperature as the controlling factor of dielectric permittivity. Aside from the temperature variations we referred to, originating on the lunar surface, there are thermal gradients originating in internal sources; these have values of less than 4°K/m (Langseth et al., 1973) and should not offset the dominating role of density in regolith depths of a few tens of meters.

Loss tangent values of sample 74241,2 indicate that variations in density produce small alterations in tan δ; a slight tendency to increase with increasing densities is shown by the values in Figure 4. The corresponding values for the terrestrial material also show a slight tendency to increase for the 100°K temperature; however, they show no particular tendency for the higher temperatures, probably owing to variable amounts of water molecules affecting the losses.

Based on data from the lunar analogue two inferences were made (Alvarez, 1973a) regarding the dielectric behavior of the lunar regolith; they were presented subject to confirmation by actual lunar material data obtained under lunarlike conditions. The first one established that in 5 to 10 cm depth the κ' values would be controlled by surface temperature variations. With the results on sample 74241,2 plus those of Carrier et al. (1973) it is now possible to refine the above prediction.

For the 1.38 g/cm^3 density value (Figure 1a), which may be considered a representative in situ density of the first two centimeters of lunar regolith, it is evident that dielectric variations will be controlled by temperature, since at such a depth the density increase is less than 0.1 g/cm^3 (Carrier et al., 1973), and for a 5 cm depth the increase is around .12 g/cm^3. Thus we can now establish that in a surface layer no more than 5 cm depth κ' variations are periodic, controlled by surface temperatures and no more than 0.2 between the maximum and minimum temperatures. At regolith depths of more than 5 cm the periodicity will disappear (i.e., due to amplitude attenuation of the temperature wave) and κ' variations will depend on density and, consequently, on depth. Loss tangent variations with temperature in the first 5 cm depth may be considered constant according to the data in Figure 1b.

CONCLUSION

Comparison of the as-received dielectric response of lunar sample 74241,2 and responses at high-vacuum conditions suggested that volatiles may have accumulated in the sample during storage and transport. The present data were insufficient to decide in which particular way volatiles alter the samples' dielectric properties; however, two possibilities were advanced: (1) physisorption of solar wind trapped gases to grain surfaces and (2) water vapor formation in submonolayer amounts.

Lunar sample values of κ' and tan δ were compared to the corresponding values in a terrestrial sample prepared to simulate lunar fines. It was noted that the κ' and tan δ variations with temperature for the lunar sample were considerably smaller than those of the terrestrial material at lunarlike conditions and frequencies below 10 KHz. κ' values of the terrestrial material at 100 KHz tended to approach the behaviour of the lunar sample. It was concluded that the terrestrial material at ultra-dried conditions would have smaller losses than the lunar sample.

Finally, it was indicated that κ' variations in the first 5 cm of lunar regolith should be periodic, controlled by surface temperature variations during the synodic period, while loss tangent values should remain approximately constant. At greater depths κ' should be controlled by density.

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FIGURE CAPTIONS

FIG. 1 (a) Dielectric permittivity against frequency and (b) loss tangent against frequency for lunar sample 74241,2. The density value is 1.38 g/cm^3; corresponding pressures and temperatures are indicated for each symbol. The temperature sequence was as indicated in the legend.

FIG. 2 (a) Dielectric permittivity against frequency and (b) loss tangent against frequency for lunar sample 74241,2. The density value is 1.61 g/cm^3; corresponding pressures and temperatures are indicated for each symbol. The temperature sequence was as indicated in the legend.

FIG. 3 Dielectric permittivity versus density for lunar sample 74241,2 and a terrestrial material simulating lunar fines, with temperature as a parameter for (a) frequency of 1 KHz. As the frequency increases the behaviour of the terrestrial material approaches that of the lunar sample.

FIG. 4 Loss tangent versus density for lunar sample 74241,2 and a terrestrial material simulating lunar fines. These data correspond to those of Figure 3. Of particular interest are the smaller losses of the terrestrial material for temperatures of 100° and 298°K.

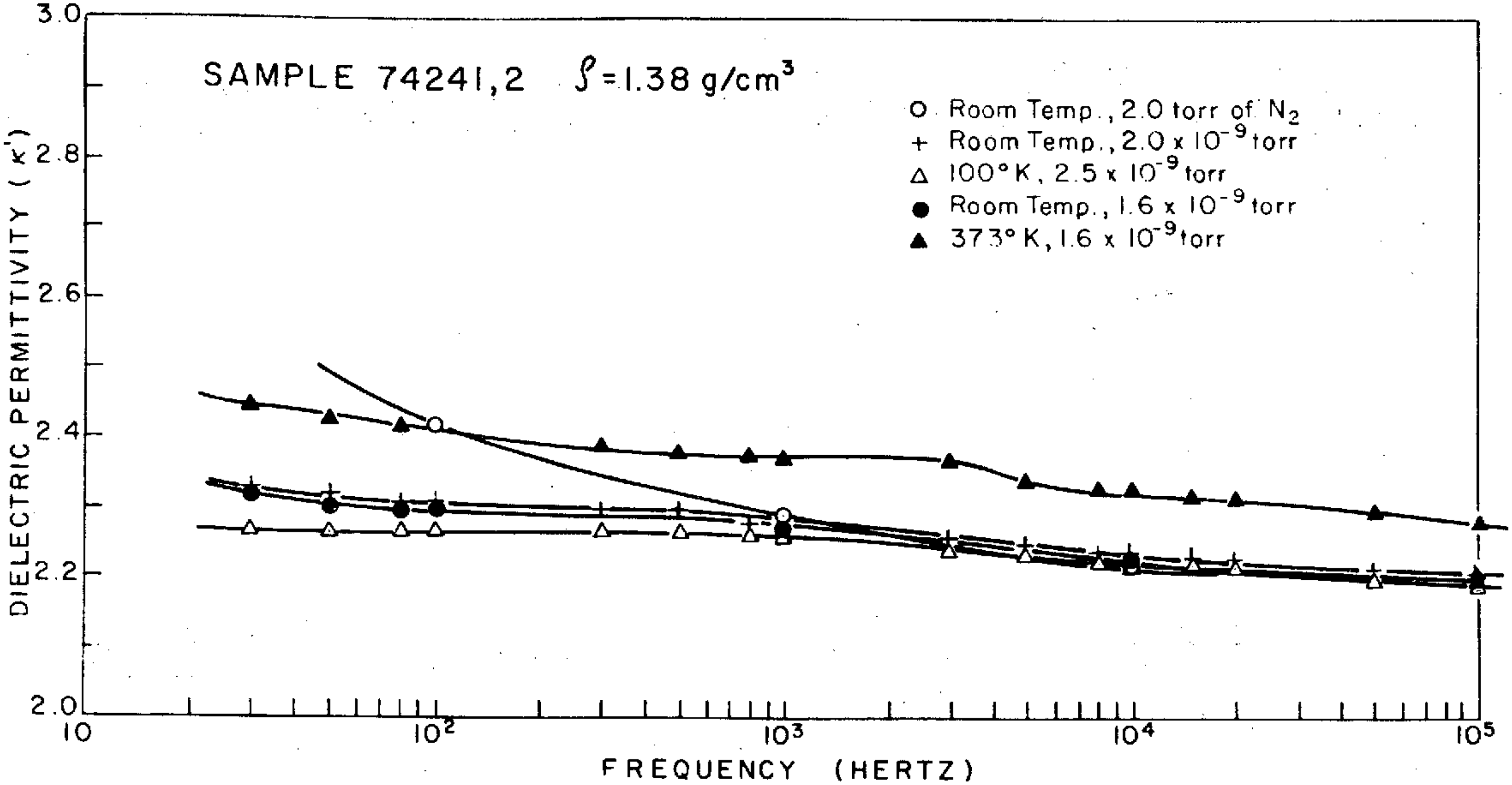

SAMPLE 74241,2 ρ=1.38 g/cm³
○ Room Temp., 2.0 torr of N₂
+ Room Temp., 2.0 x 10⁻⁹ torr
△ 100°K, 2.5 x 10⁻⁹ torr
● Room Temp., 1.6 x 10⁻⁹ torr
▲ 373°K, 1.6 x 10⁻⁹ torr
DIELECTRIC PERMITTIVITY (κ')
3.0
2.8
2.6
2.4
2.2
2.0
10
10²
10³
10⁴
10⁵
FREQUENCY (HERTZ)

Figure 1a

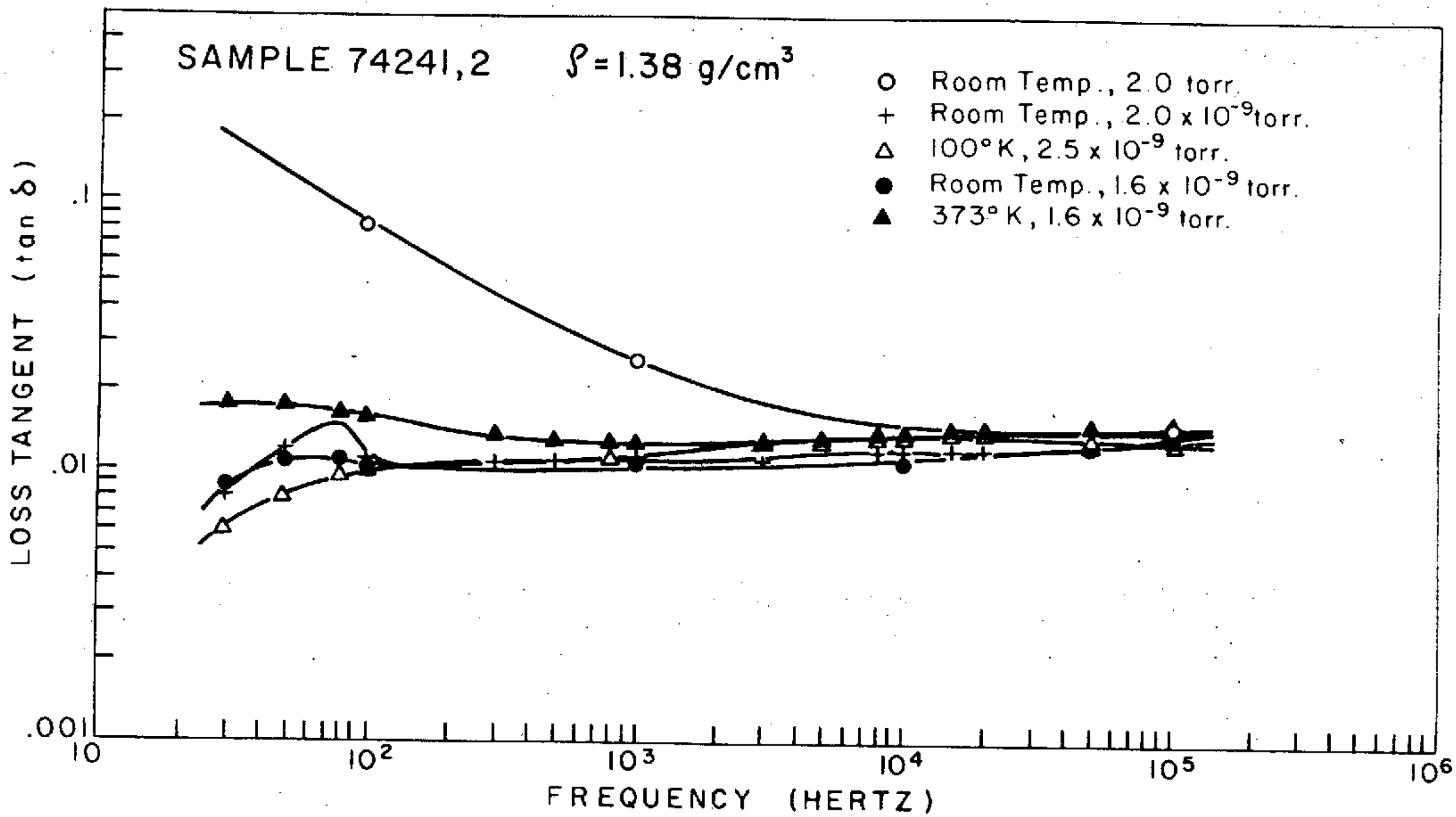
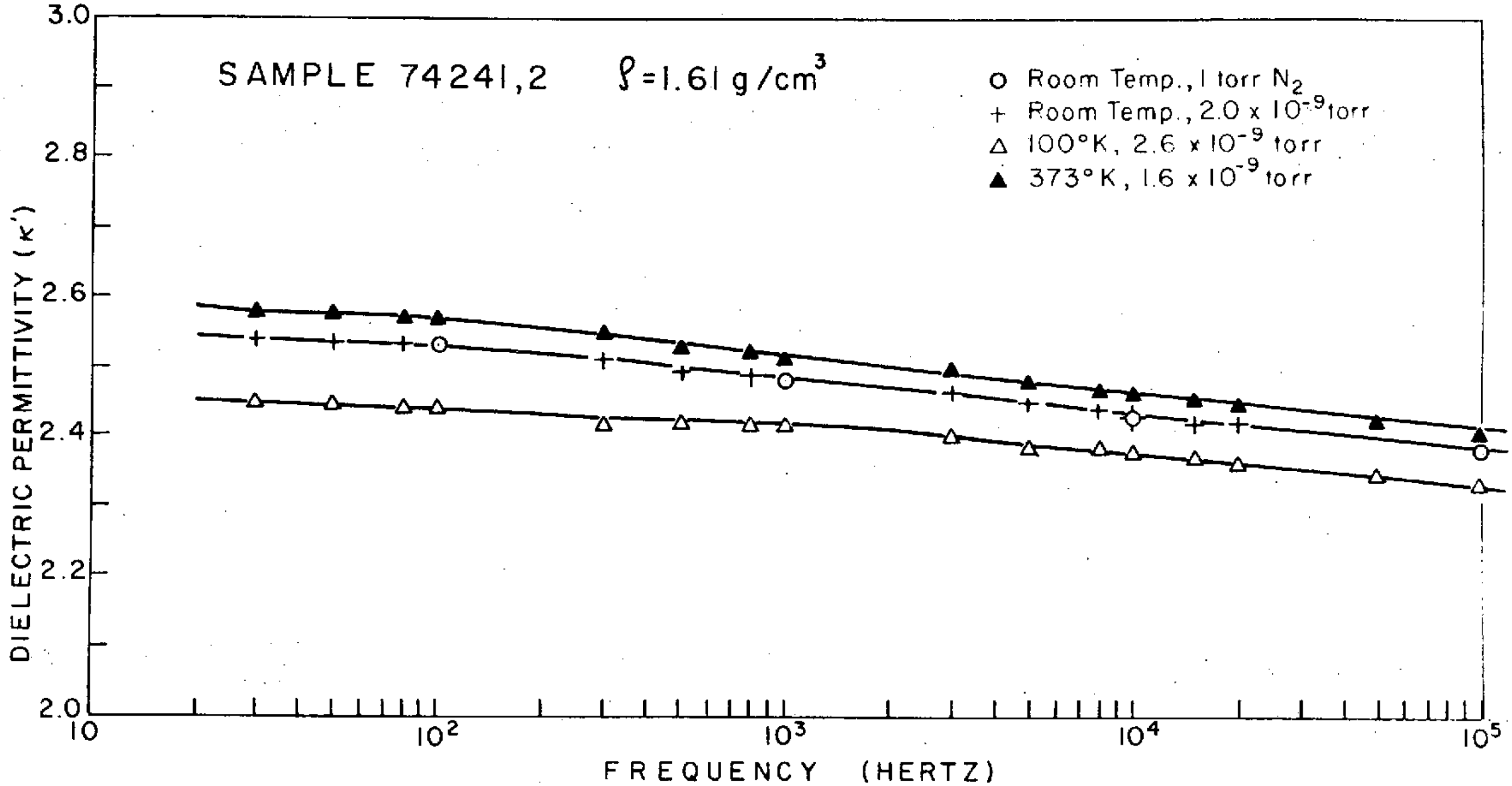
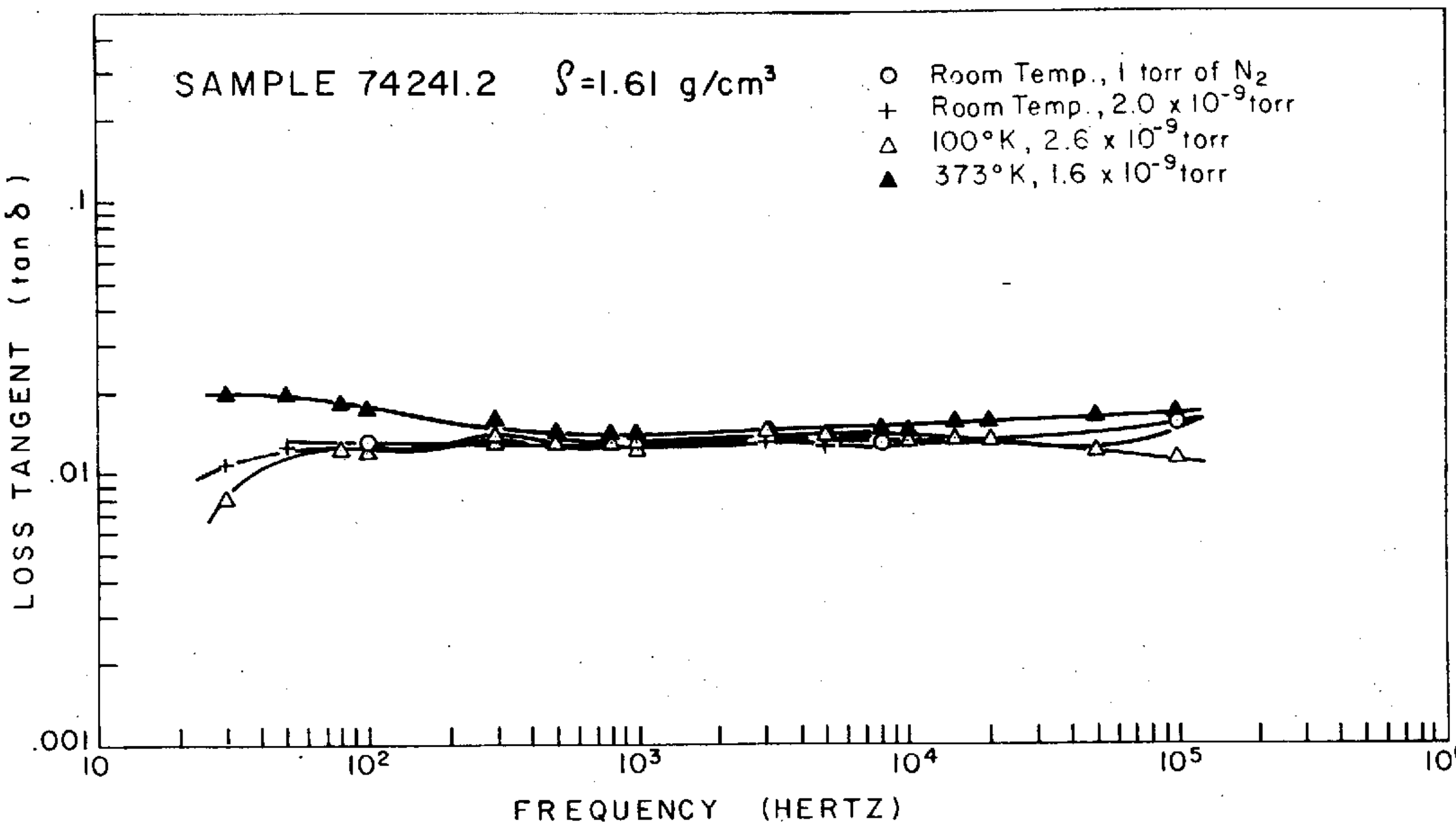

SAMPLE 74241,2 ρ=1.38 g/cm³
Room Temp., 2.0 torr
Room Temp., 2.0 x 10⁻⁹ torr.
100°K, 2.5 x 10⁻⁹ torr.
Room Temp., 1.6 x 10⁻⁹ torr.
373°K, 1.6 x 10⁻⁹ torr.
LOSS TANGENT (tan δ)
.1
.01
.001
10
10²
10³
10⁴
10⁵
10⁶
FREQUENCY (HERTZ)

Figure 1b



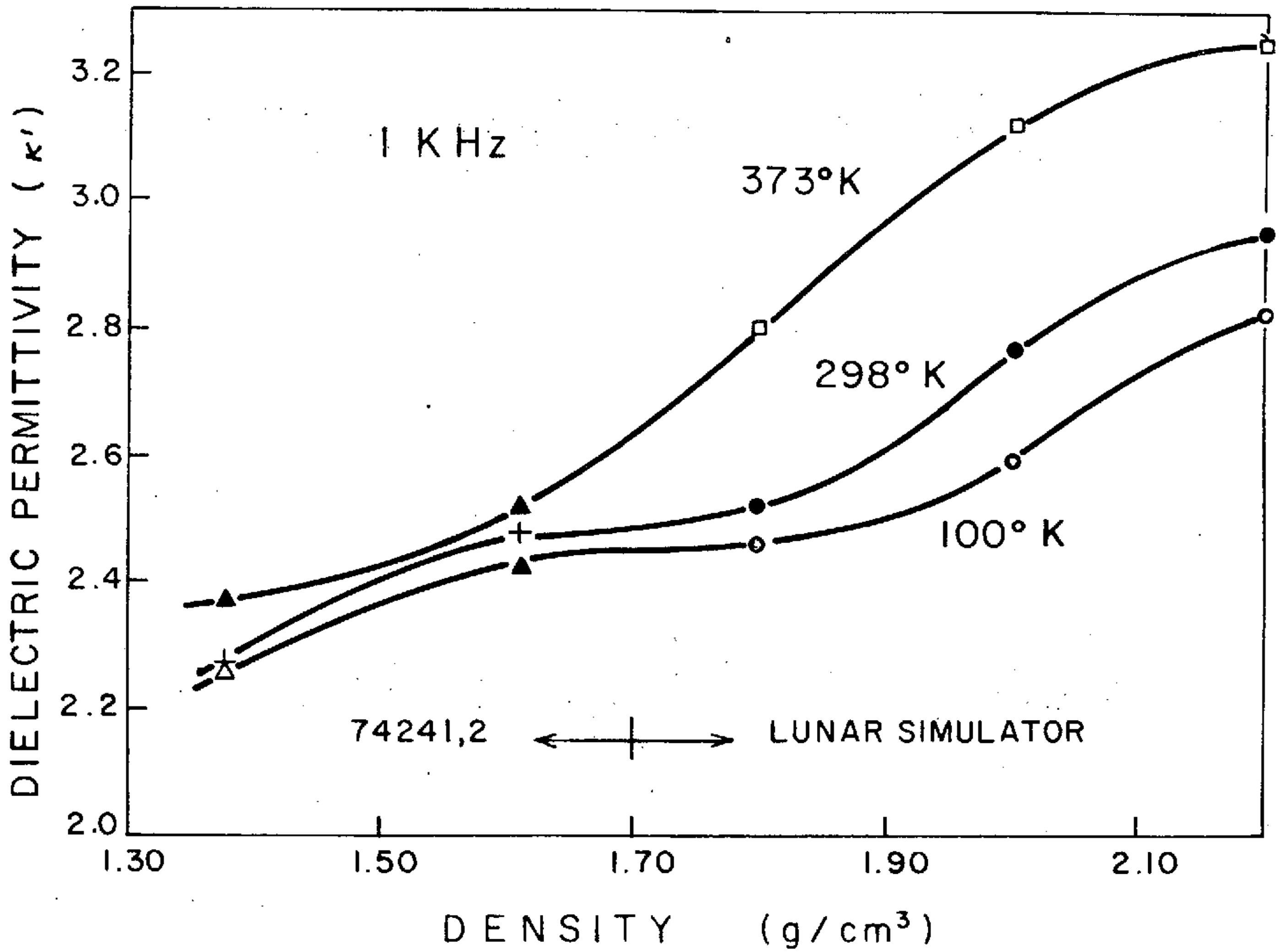
SAMPLE 74241,2 ρ=1.61 g/cm³
○ Room Temp., 1 torr N₂
+ Room Temp., 2.0 x 10⁻⁹ torr
△ 100°K, 2.6 x 10⁻⁹ torr
▲ 373°K, 1.6 x 10⁻⁹ torr
DIELECTRIC PERMITTIVITY (κ')
3.0
2.8
2.6
2.4
2.2
2.0
10
10²
10³
10⁴
10⁵
FREQUENCY (HERTZ)

Figure 2a



SAMPLE 74241.2 ρ=1.61 g/cm³
Room Temp., 1 torr of N_2
Room Temp., 2.0×10^{-9} torr
100°K, 2.6×10^{-9} torr
373°K, 1.6×10^{-9} torr
LOSS TANGENT (tan δ)
.1
.01
.001
10
10^2
10^3
10^4
10^5
10^6
FREQUENCY (HERTZ)

Figure 2b



1 KHz
373°K
298° K
100° K
74241,2
LUNAR SIMULATOR
DIELECTRIC PERMITTIVITY (κ')
DENSITY (g/cm³)
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.30
1.50
1.70
1.90
2.10

Figure 3a

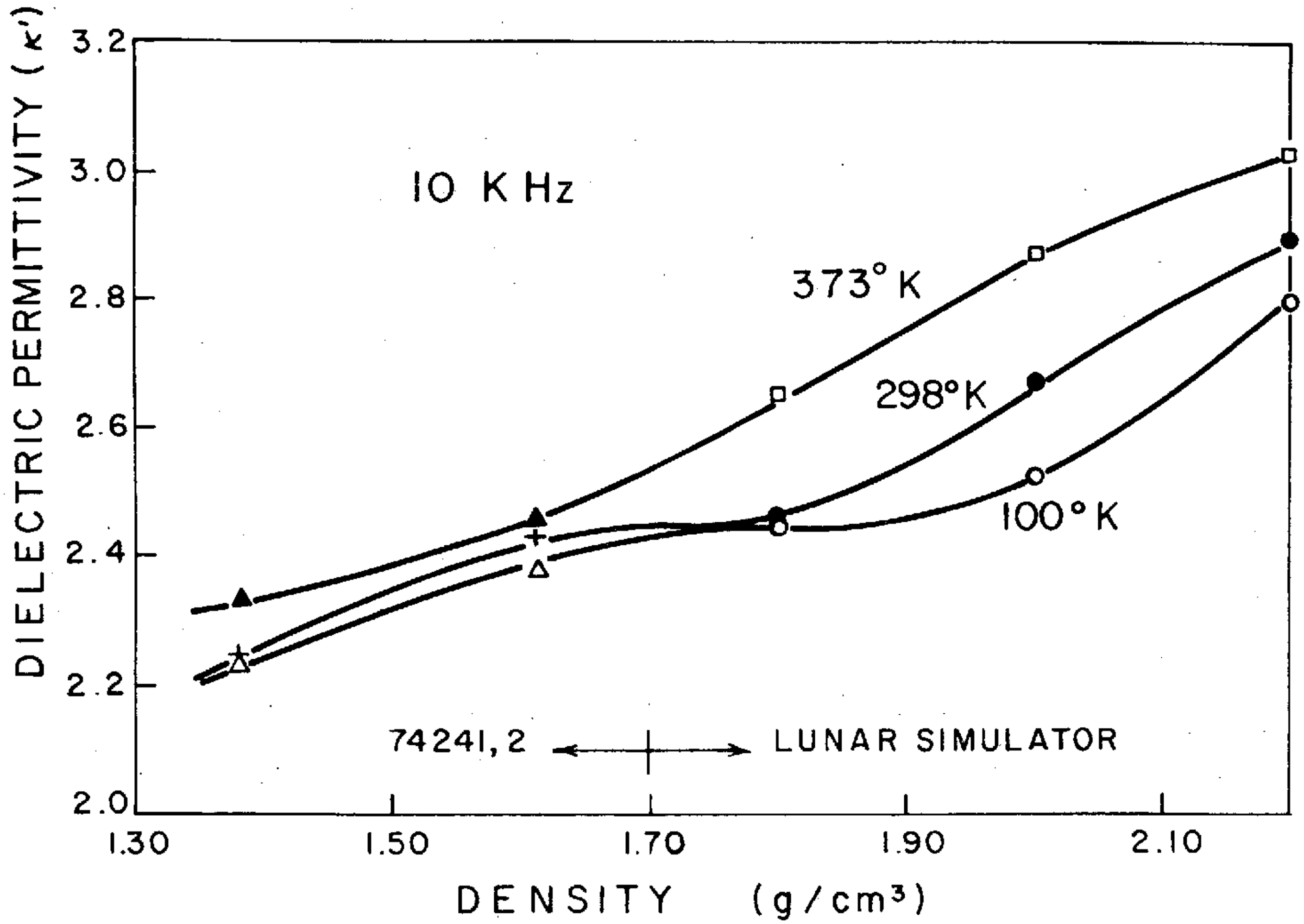

DIELECTRIC PERMITTIVITY (κ')
3.2
3.0
2.8
2.6
2.4
2.2
2.0
10 KHz
373° K
298°K
100° K
74241,2
LUNAR SIMULATOR
1.30
1.50
1.70
1.90
2.10
DENSITY (g/cm³)

Figure 3b

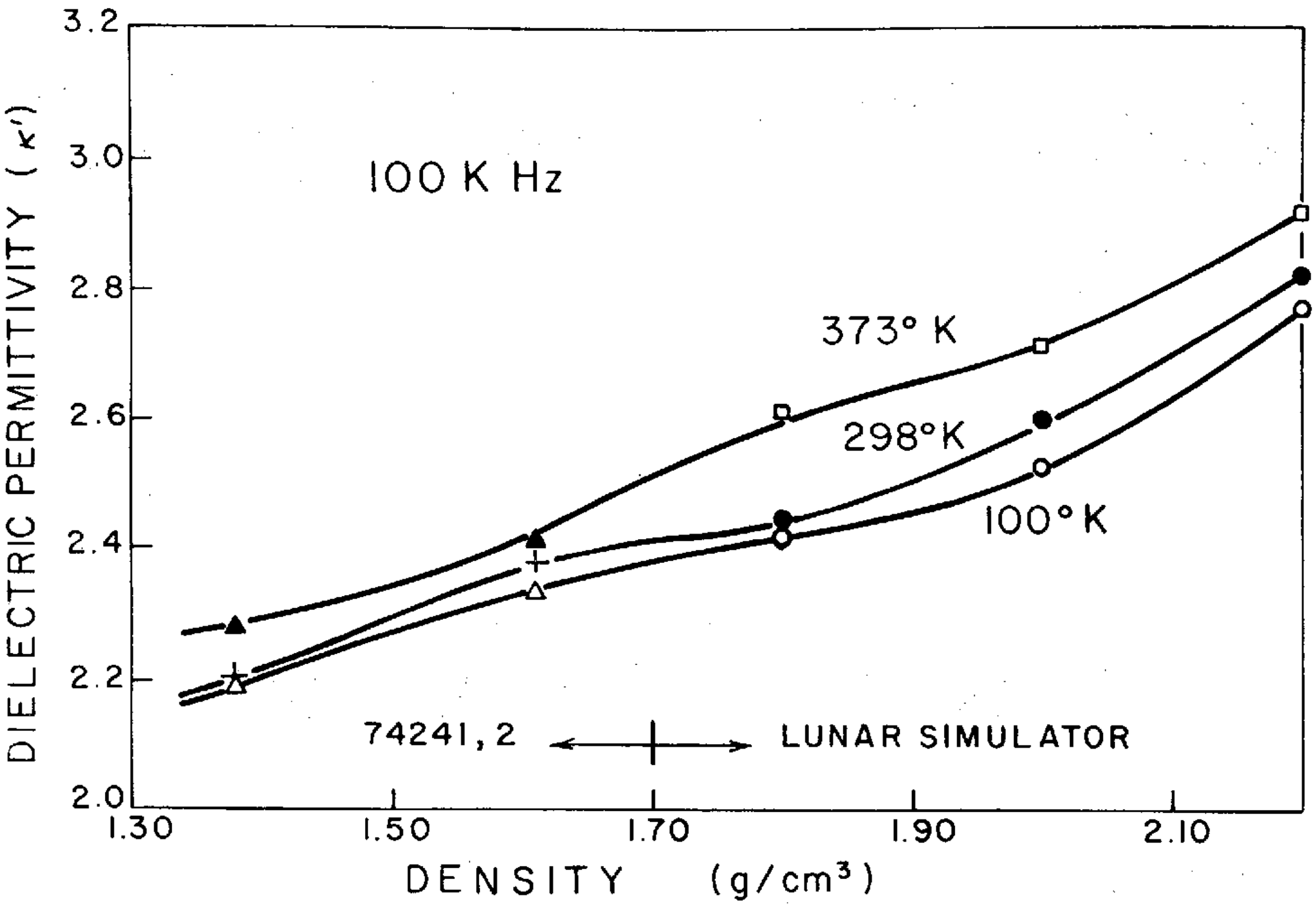
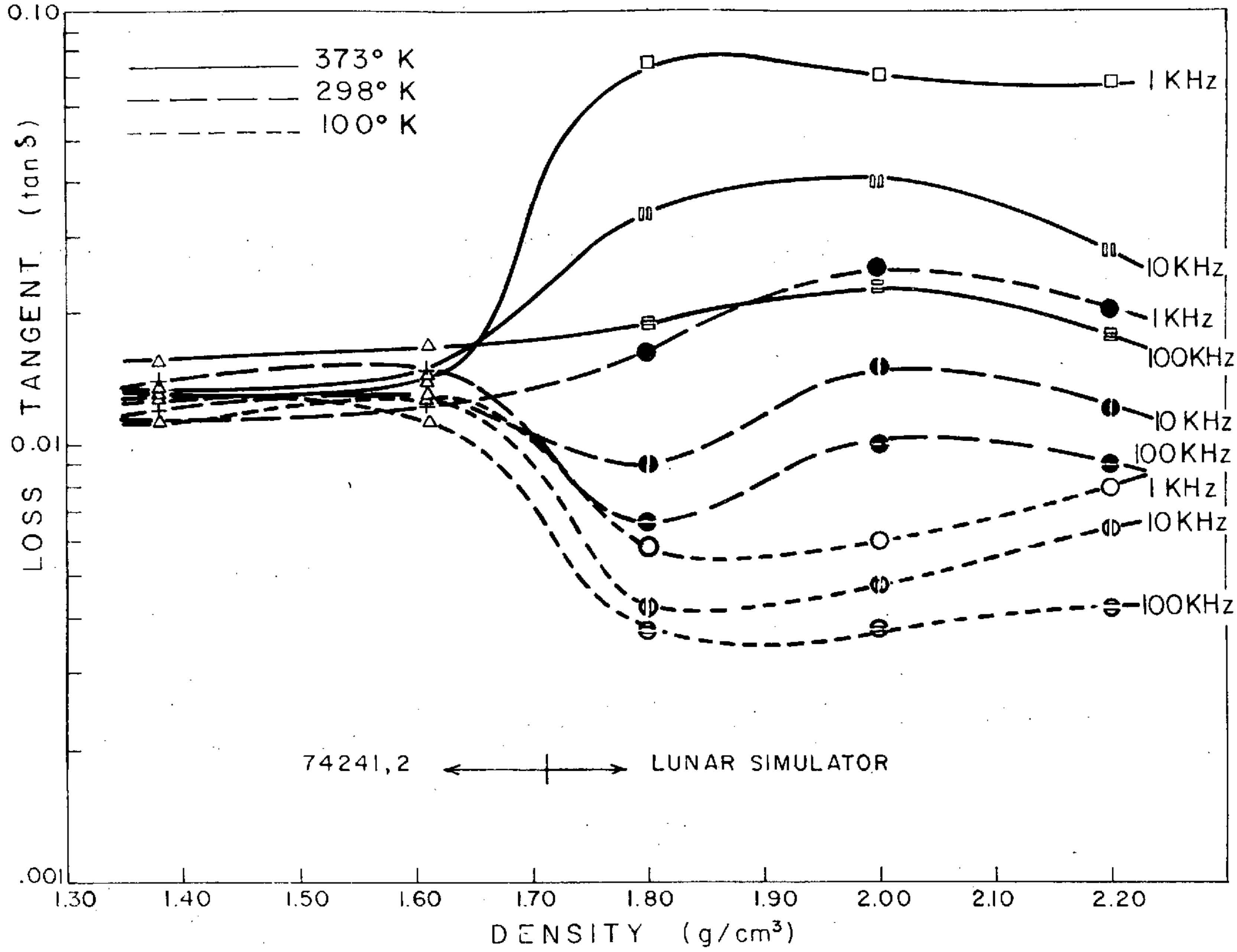

100 K Hz
373° K
298°K
100° K
74241, 2
LUNAR SIMULATOR
DIELECTRIC PERMITTIVITY (κ')
DENSITY (g/cm³)
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.30
1.50
1.70
1.90
2.10

Figure 3c



0.10
0.01
.001
373° K
298° K
100° K
1 KHz
10 KHz
1 KHz
100 KHz
10 KHz
100 KHz
1 KHz
10 KHz
100 KHz
LOSS TANGENT (tan δ)
74241,2
LUNAR SIMULATOR
1.30
1.40
1.50
1.60
1.70
1.80
1.90
2.00
2.10
2.20
DENSITY (g/cm³)

Figure 4

PART II

ELECTRICAL PROPERTIES OF SAMPLE 70215 IN THE TEMPERATURE RANGE OF 100° to 373°K

Dielectric permittivity, loss tangent, and dc conductivity of solid sample 70215,14 have been measured at temperatures of 100°, 298°, 315°, and 373°K. Measurements were performed at a pressure of 1 torr of N_2 or in vacuums of $\sim 10^{-7}$ to 10^{-8} torr; the sample was never exposed to the atmosphere. Dielectric properties were obtained in the 30 Hz to 100 KHz frequency range. The basic experimental procedure has been reported elsewhere (Alvarez, 1973a). In comparison with other lunar material this sample presents a rather high dc conductivity. Sample 70215 has been described (Lunar Sample Info. Cat., Apollo 17, 1973) as a fine-grained basalt with a groundmass (48% of rock) of Ilmenite (?), Pyroxene, and Plagioclase, and phenocrysts (52% of rock) of Ilmenite (?), Olivene, and Pyroxene. The results obtained may help characterize materials in the regolith and in the upper layers of the lunar basement.

Figure 1a shows the dielectric permittivity data. The Temperature sequence is indicated in the legend of the figure; the corresponding information for tan δ appears in Figure 1b. Values of dielectric permittivity at 100 KHz vary from 6.25 at 100°K to 8.18 at 373°K; the lower frequencies, however, show a considerably larger scatter with temperature. This behaviour is typical of dielectrics with non-negligible ohmic conductivities, in contact with metallic electrodes (Alvarez, 1973b). Clustering of the data at the higher frequencies suggests that such an effect becomes negligible above 100 KHz and, thus, representative values of κ' may be taken at this frequency for temperatures of 298°K and above. The data at 100°K appears to be free of dc conductivity effects; consequently, all reported values are considered representative of the sample properties at this temperature. Tan δ variations do not exceed one order of magnitude in the temperature range analyzed.

Figure 2 is a plot of dc conductivity versus the reciprocal temperature with applied voltage as a parameter. Based on the data at the higher temperatures we have assumed a region of linear variation (thick, straight line), from which an apparent energy gap of 0.53 eV has been computed. If the sample were a crystalline semiconductor, this temperature region would correspond to an intrinsic conduction; however, from the sample composition it is obvious that we are dealing with a multicrystalline system, possibly to be regarded as an amorphous semiconductor, and we must refrain from labeling such a region as of intrinsic conduction.

The behaviour in the low-temperature region would suggest a change to extrinsic conduction (i.e., by impurities) if the sample were a crystalline specimen; however, it could also be explained in terms of thermally assisted tunnelling, after the conduction model for amorphous semiconductors proposed by Davis and Mott (1970). Unfortunately, only one conductivity value was obtained in the low-temperature region; more data at low temperatures is needed to pin down the actual electrical classification of the sample. The dashed lines indicate only a possible behaviour between room temperature and 77°K (i.e., between values of 3.35 and 12.98 for 1000/T°K); they may be in great discrepancy from actual conductivity values in such a region. The sample presents a non-ohmic behaviour in the temperature range analyzed; it is evidenced by increasing dc conductivities with increasing applied voltages. Conductivity values for voltages of 500 and 1000 volts, and temperature of 373°K were greater than 10^{-7} $(\Omega\text{-m})^{-1}$; they were not determined owing to limitations in the measuring range of the resistivity meter.

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Davis, E.A., and N.F. Mott, "Conduction in Non-crystalline Systems." "Conductivity, Optical Absorption and Photoconductivity in Amorphous Semiconductors," Phil. Mag., 22, 903, 1970.

Lunar Sample Information Catalog, Apollo 17, NASA document MSC 03211, pp. 133-136, 1973.

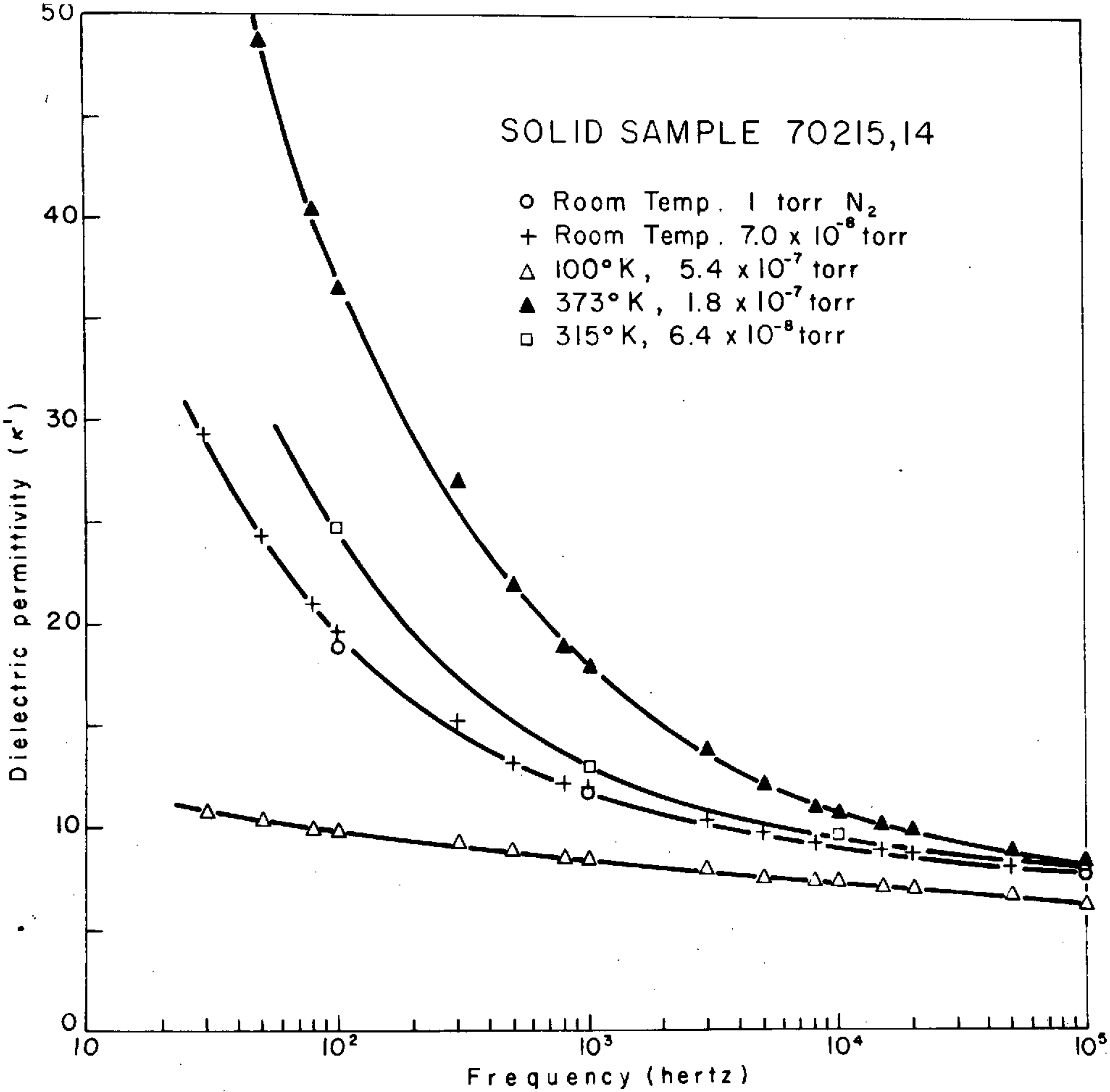
SOLID SAMPLE 70215,14
○ Room Temp. 1 torr N2
+ Room Temp. 7.0 x 10-8 torr
△ 100° K, 5.4 x 10-7 torr
▲ 373° K, 1.8 x 10-7 torr
□ 315° K, 6.4 x 10-8 torr
Dielectric permittivity (κ')
50
40
30
20
10
0
10
10^2
10^3
10^4
10^5
Frequency (hertz)

Figure 1a

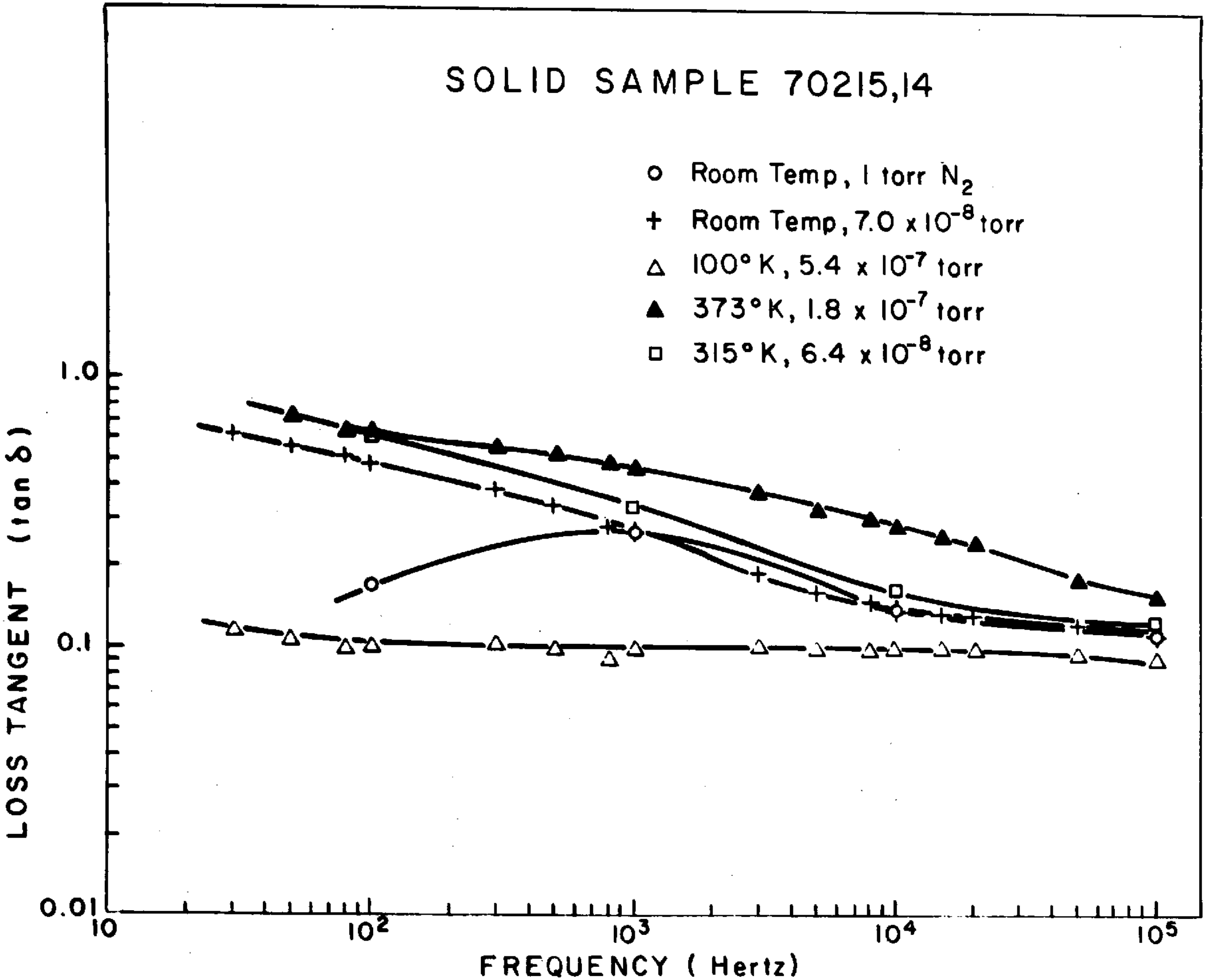
SOLID SAMPLE 70215,14
Room Temp, 1 torr N_2
Room Temp, 7.0 x 10^{-8} torr
100° K, 5.4 x 10^{-7} torr
373° K, 1.8 x 10^{-7} torr
315° K, 6.4 x 10^{-8} torr
LOSS TANGENT (tan δ)
1.0
0.1
0.01
10
10^2
10^3
10^4
10^5
FREQUENCY (Hertz)

Figure 1b

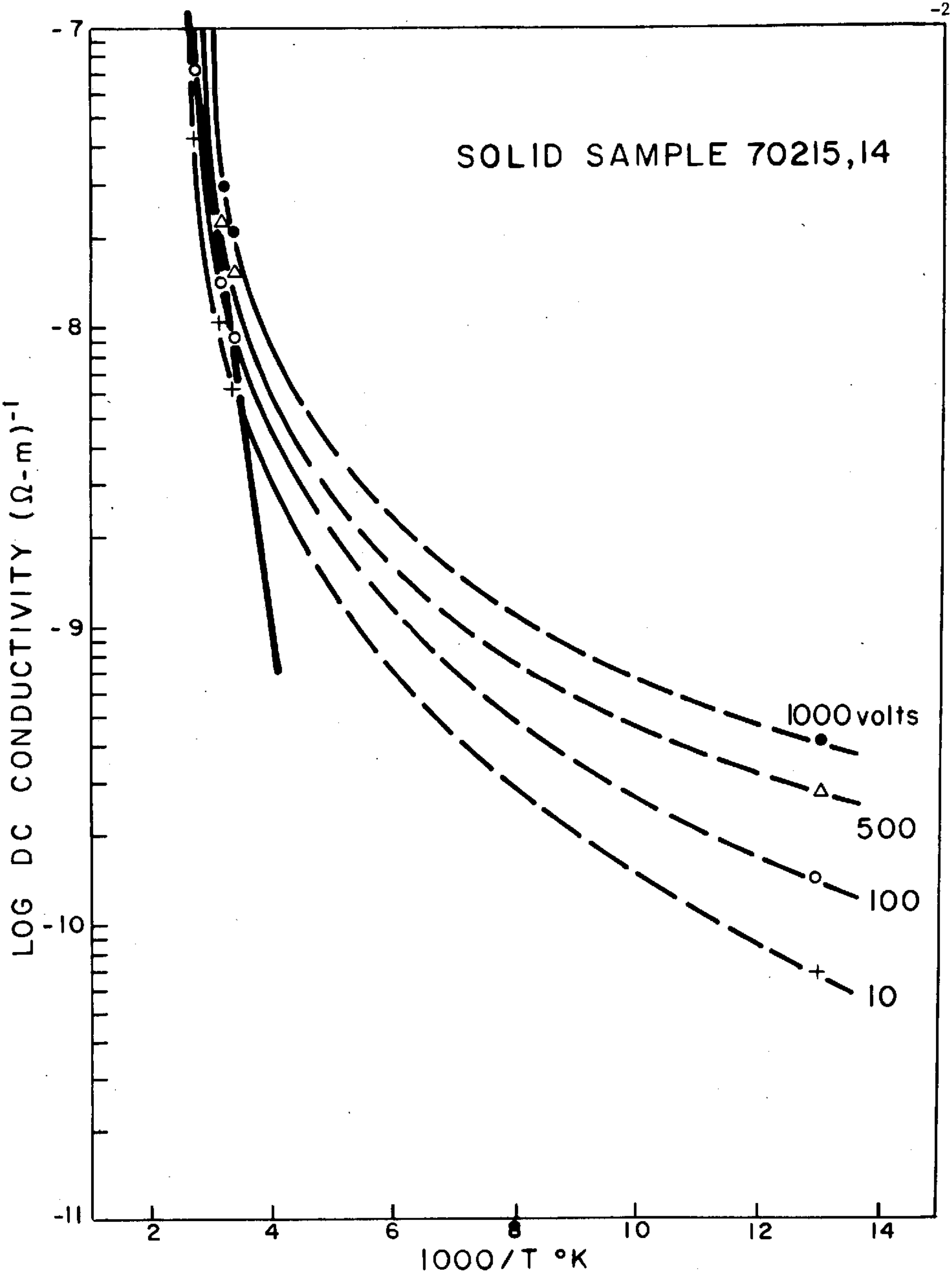
SOLID SAMPLE 70215,14
LOG DC CONDUCTIVITY (Ω-m)-1
-7
-8
-9
-10
-11
2
4
6
8
10
12
14
1000/T °K
1000 volts
500
100
10

Figure 2